METHOD 7521

NICKEL (ATOMIC ABSORPTION, FURNACE METHOD)

1.0 SCOPE AND APPLICATION

1.1 See Section 1.0 of Method 7000.

2.0 SUMMARY OF METHOD

2.1 See Section 2.0 of Method 7000.

3.0 INTERFERENCES

3.1 If interferences are suspected, see Section 3.0 of Method 7000.

3.2 In addition to the normal interferences experienced during graphite furnace analysis, nickel analysis can suffer from severe nonspecific absorption and light scattering caused by matrix components during atomization. Background correction is strongly recommended.

3.3 Severe memory effects for nickel may occur in graphite furnace tubes used for arsenic or selenium analysis by Methods 7060 and 7740, resulting from the use of a nickel nitrate matrix modifier in those methods. Use of graphite furnace tubes and contact rings for nickel analysis that are separate from those used for arsenic and selenium analyses is strongly recommended.

4.0 APPARATUS AND MATERIALS

4.1 For basic apparatus, see Section 4.0 of Method 7000. Due to the widespread use of a nickel-nitrate modifier for atomic absorption analyses, a dedicated instrument is recommended when conducting analyses by this method. If a dedicated instrument is not available, the furnace tubes and contact rings should be changed prior to using this methodology.

4.2 Instrument parameters (general):

4.2.1 Drying time and temp.: 30 sec at 125°C.

4.2.2 Ashing time and temp.: 30 sec at 800°C.

4.2.3 Atomizing time and temp.: 10 sec at 2700°C.

4.2.4 Purge gas: Argon or nitrogen.

4.2.5 Wavelength: 232.0 nm.

4.2.6 Background correction: Recommended.

4.2.7 The above operating parameters and any others should be set as specified by the particular instrument manufacturer.

NOTE: The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-µL injection, continuous-flow purge gas, and nonpyrolytic graphite. Smaller size furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above-recommended settings.

5.0 REAGENTS

5.1 See Section 5.0 of Method 7000.

5.2 Preparation of standards

5.2.1 Stock solution - Do not dry reagent. Dissolve 4.953 g of nickel nitrate hexahydrate, $Ni(NO_3)_2 \bullet 6H_2O$ (analytical reagent grade) in reagent water and dilute to 1.000 L in a volumetric flask. Alternatively, procure a certified standard from a supplier and verify concentration by comparison with a second standard.

5.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The calibration standards should be prepared using the same type of acid and at the same acid concentration as in the prepared samples to be analyzed (0.5% v/v HNO_3).

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See Chapter Three, Section 3.1.3, "Sample Handling and Preservation."

7.0 PROCEDURE

7.1 Sample preparation - The procedures for preparation of the sample are given in Chapter Three, Section 3.2.

7.2 See Method 7000, Section 7.3, "Furnace Technique."

8.0 QUALITY CONTROL

8.1 See Section 8.0 of Method 7000.

9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are not available at this time.

9.2 The performance characteristics for an aqueous sample free of interferences are as follows:

Optimum concentration range: 5-50 µg/L.
Estimated detection limit: 1 µg/L.

10.0 REFERENCES

1. Methods for the Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory, ORD Publication Offices of the Center for Environmental Research Information, Cincinnati, OH, 1983; EPA-600/4-79-020.

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